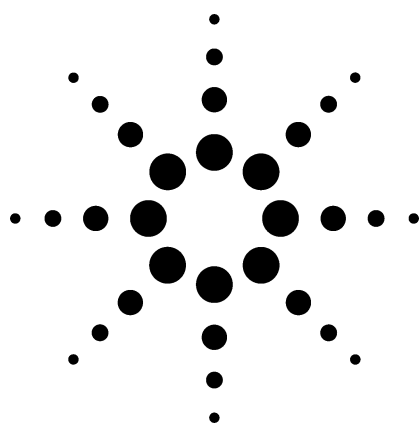


Applications of Retention Time Locking in Simulated Distillation



Application

Gas Chromatography

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Abstract

Retention time locking is used to lock retention times when transferring methods between chromatographic instruments, between columns, and when changing to a different type of detector. The retention times for a simulated distillation calibration standard analyzed on a second GC system matched the original retention times on the first system within 0.02 minutes after locking. Thus, using retention time locking, retention time reproducibility between GC systems meets typical ASTM simulated distillation specifications for reproducibility on a single system.

Key Words

Retention time locking (RTL), method validation, ASTM D 2887, capillary gas chromatography, laboratory productivity, SimDis, simulated distillation.

Introduction

Simulated distillation (SimDis) is one of the most common analyses in the petroleum industry. Many laboratories using SimDis run duplicate methodology on several instruments. However, the retention times for each instrument differ slightly, which means that each instrument must have a separate calibration and integration event table. Differences in retention times also complicate comparison of data between instruments and over time. The short, large-diameter columns used for higher-boiling ("extended") SimDis are particularly prone to retention time differences due to the very thin stationary phase and low pressure drop.

Retention time locking (RTL) is the ability to match chromatographic retention times exactly in any system

to those in another chromatographic system, with the same nominal column. Agilent RTL software allows rapid, accurate locking of all retention times. This application note examines the use of retention time locking with RTL software for a SimDis sample run according to an extended ASTM D 2887¹, which extends the ASTM method to include analysis of hydrocarbons boiling up to n-C₆₀.

Experimental

An Agilent 6890 Series gas chromatograph (GC) with electronic pneumatics control (EPC) was used for these experiments. Sample injection was accomplished with an Agilent G1916A automatic liquid sampler (ALS). The 6890 was equipped with a cool on-column inlet and a flame ionization detector (FID). A 10 m x 0.53 mm, 0.88 μ m HP-1 column (part number 19095Z-021) was used for separation. An Agilent ChemStation was used for instrument control and data acquisition. The experimental conditions for the GC methods are given by each chromatogram.



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Results and Discussion

Figure 1 shows a SimDis sample run according to an extended ASTM D 2887 procedure. The top chromatogram represents the n-paraffin calibration mixture containing C_5 – C_{60} hydrocarbons. The bottom chromatogram is of an actual sample.

The same analysis was run under identical conditions on a new system (GC system 2), and the chromatograms are compared in figure 2. The retention times are shifted 0.15 to 0.3 minutes, which is typical when changing columns or systems.

After adjusting the inlet pressure of GC system 2 using Agilent RTL software, the retention time deviation was only 0.04 to 0.08 minutes (figure 3). The retention times for GC system 2 now meet ASTM D 2887 criteria for retention time on a single system, let alone two different systems.

After a second iteration of inlet pressure adjustment, retention times match even better as shown in figure 4. The retention times locked within 0.02 minutes.

The most accurate results for SimDis analyses would still be obtained from standard calibrations on a given system. However, these RTL experiments demonstrate that using a common (universal) boiling point calibration curve based on locked GC systems can give results that easily meet ASTM criteria for a large number of locked systems. This, in turn, would allow easier review of data from multiple instruments and over extended periods of time. This capability clearly saves time, increases the value of the data, and provides more useful historical records on sample results.

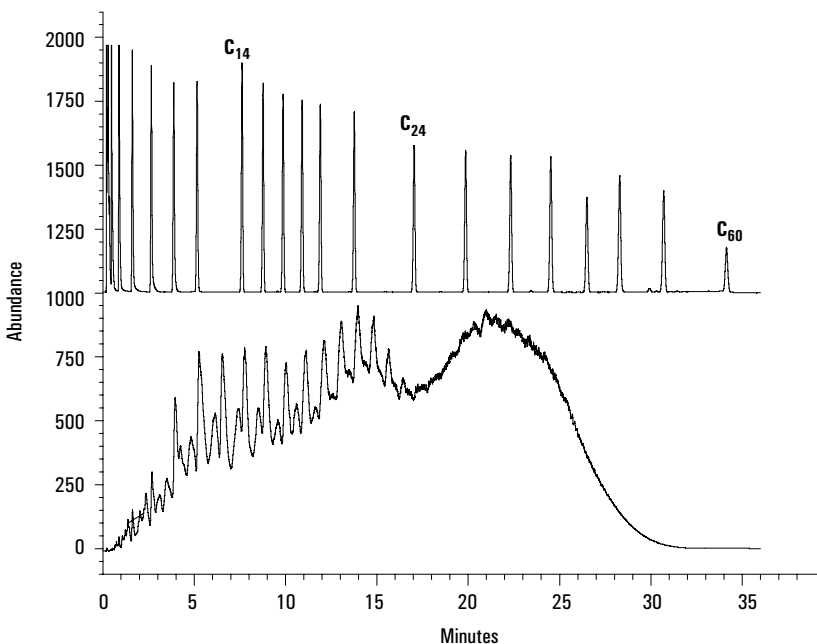


Figure 1. GC Simulated Distillation, C_5 – C_{60} range. Conditions: column, 10 m x 0.53 mm, 0.88 μ m HP-1; carrier gas: 19 mL/min helium, 4.3 psi constant flow; injection: 0.1 μ L on-column; oven program: 35 $^{\circ}$ C to 375 $^{\circ}$ C, 10 $^{\circ}$ C/min.

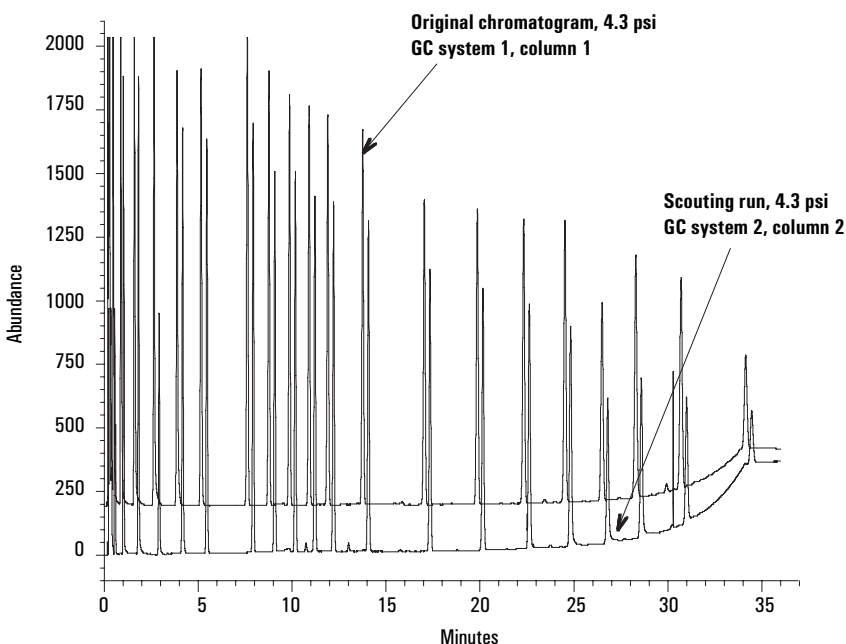


Figure 2. Comparison of the original chromatogram and a scouting run on GC System 2. The retention time shift is 0.15–0.30 minutes.

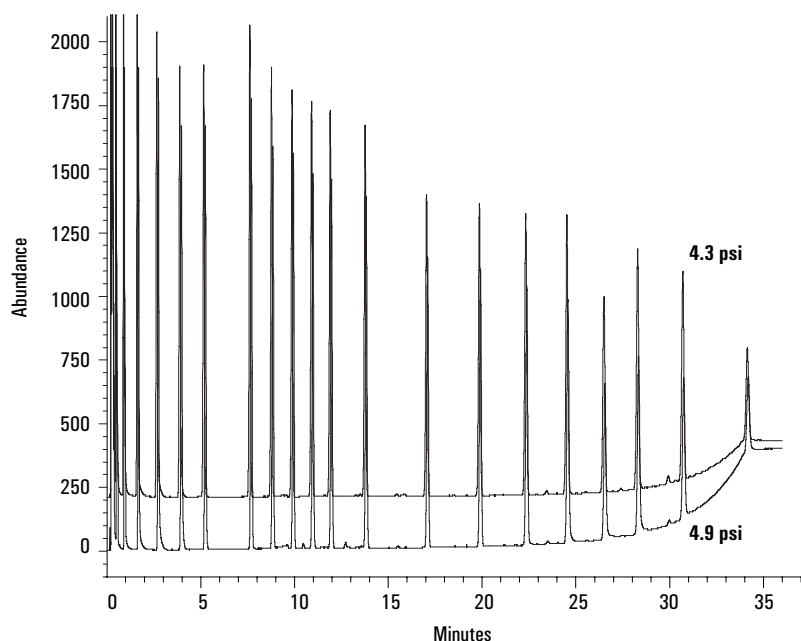


Figure 3. Comparison of original chromatogram and chromatogram obtained on GC System 2 after pressure adjustment based upon retention time vs. pressure calibration. The retention time differences between the two systems are 0.04–0.08 minutes.

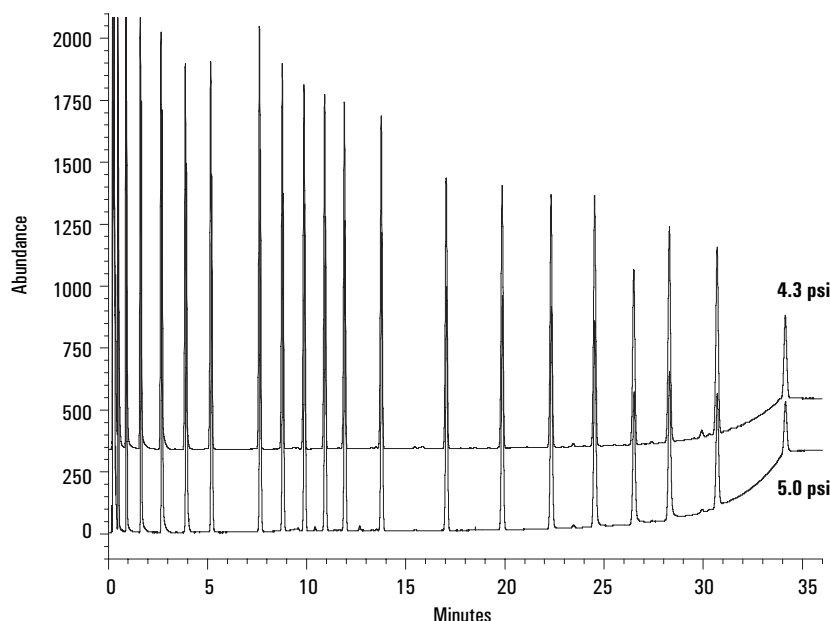


Figure 4. Comparison of original chromatogram and chromatogram obtained on GC System 2 after a second pressure adjustment based upon retention time results obtained from the first pressure correction. The largest retention time deviation between the two systems is now 0.02 minutes.

Conclusions

The Agilent RTL software was used to lock retention times for a simulated distillation sample analyzed on a second GC system. The retention times on the second system matched the original retention times on the first system within 0.02 minutes, meeting ASTM specifications for reproducibility on one system. This not only demonstrates the exceptional retention time reproducibility of the 6890 Series GC, but also the potential of RTL to improve quality, productivity, and intercomparability of capillary GC analyses.

References

1. ASTM D 2887-93, "Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography," *Annual Book of Standards*, Volume 05.02, ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428 USA.

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